Aug. 29, 1972 K. E. PORTER ET AL 3,687,818

DISTILLATION APPARATUS

Filed Feb. 3, 1970 3 Sheets-Sheet 1

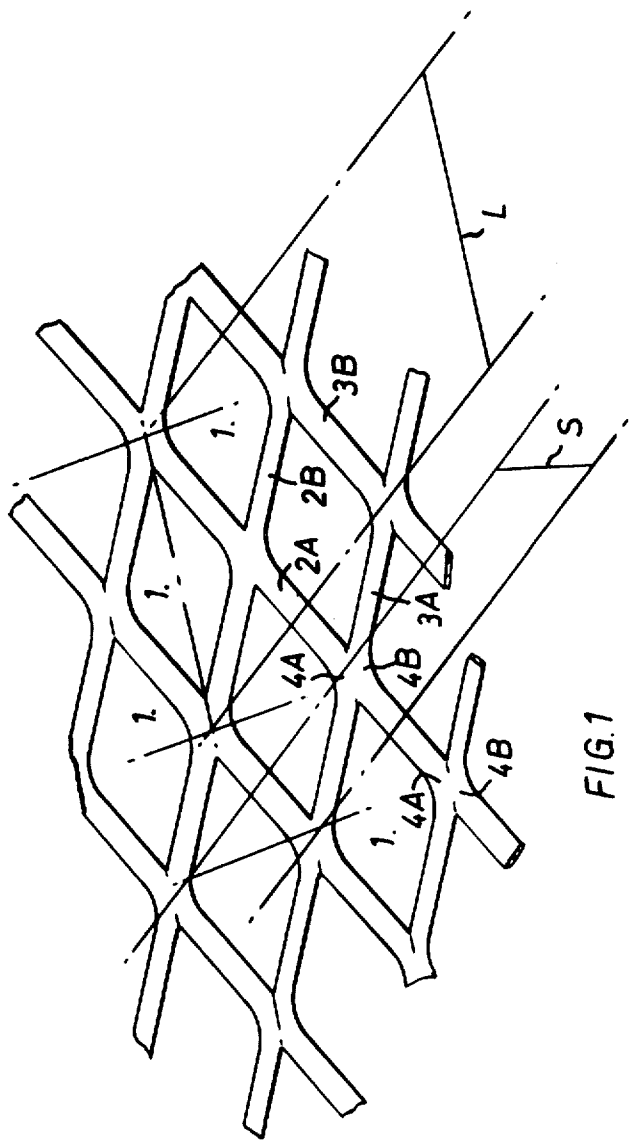



FIG.1

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DISTILLATION APPARATUS

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FIG. 2

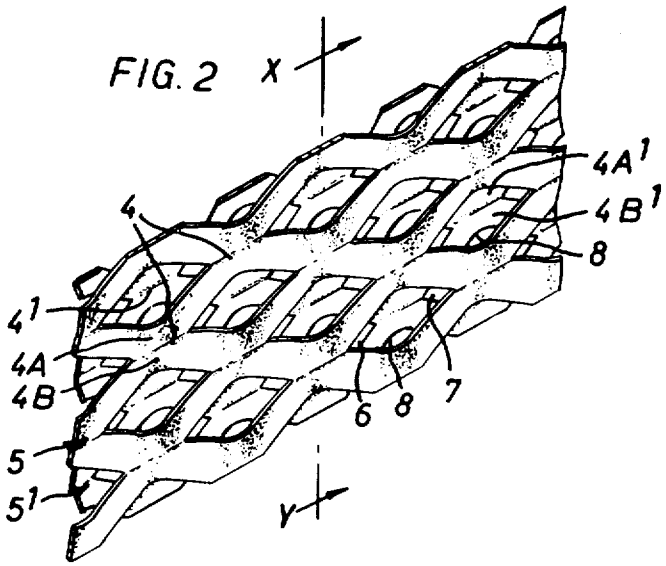
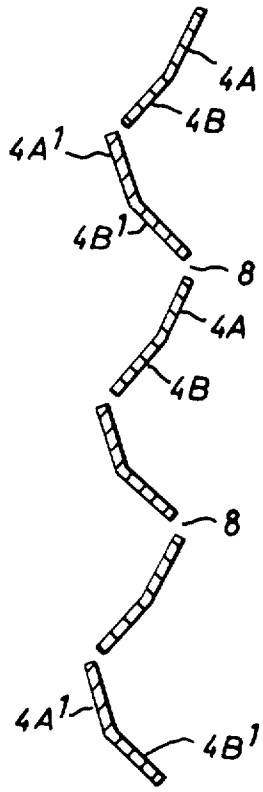


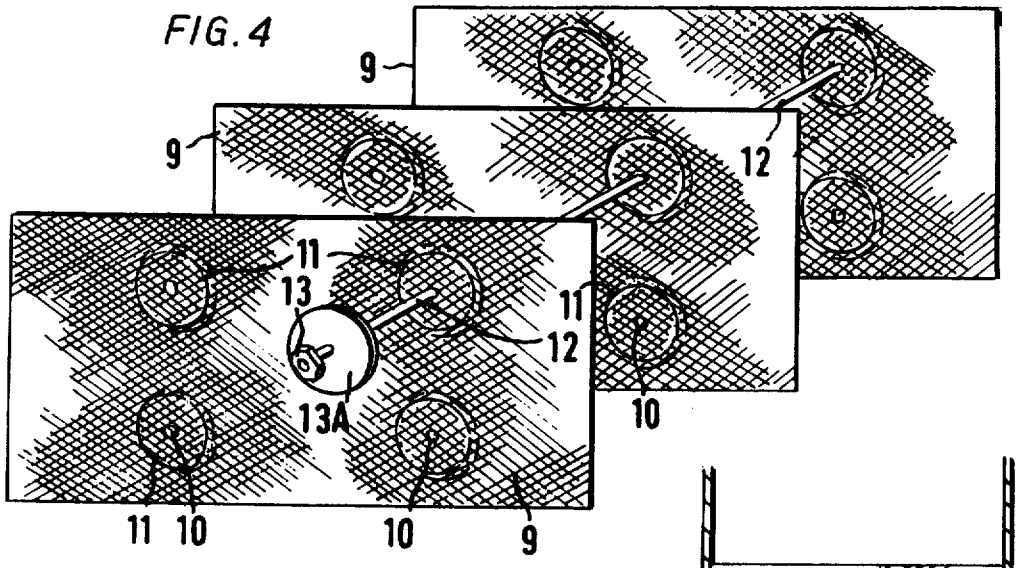

FIG. 3



DISTILLATION APPARATUS

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FIG. 4



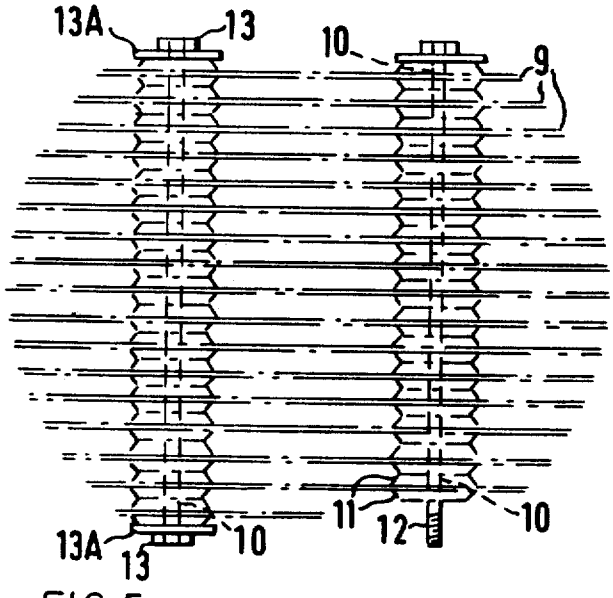




FIG. 5

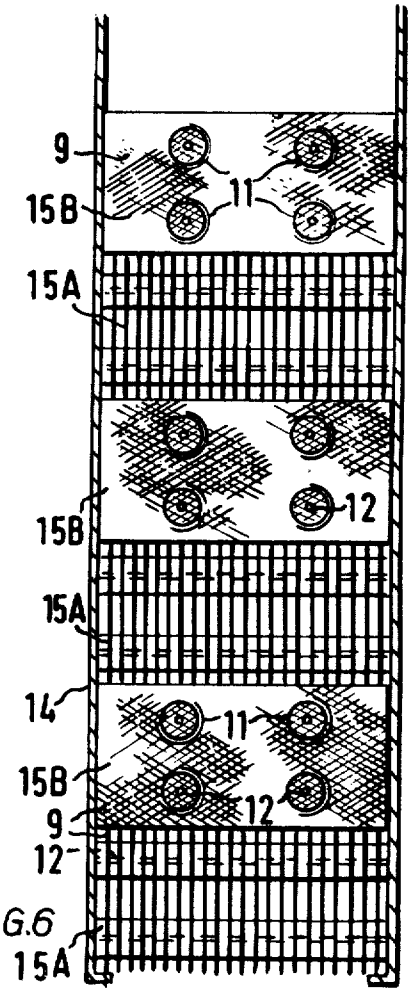



FIG. 6

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3,687,818

# DISTILLATION APPARATUS

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Int. Cl. B01d 3/14; F28f 3/08
U.S. Cl. 202—158 5 Claims

## ABSTRACT OF THE DISCLOSURE

A packing for a distillation column especially useful in low pressure distillations consists of a plurality of plates each comprising two sheets of expanded metal placed back to back in a staggering configuration.

This invention relates to distillation apparatus. More specifically it relates to a novel packing material for distillation columns which is especially suitable for use in distillation at subatmospheric pressure.

In distillation practice the need often arises to distil a substance at a temperature below its atmospheric boiling point. This is accomplished by conducting the distillation at an appropriate subatmospheric pressure.

However, in such an operation it is necessary that the packing material used in the column should not be so resistant to gas flow that it is impossible to remove distillate at an acceptable rate without raising the pressure at the bottom of the still, and hence also the temperature of its contents, above the stipulated maxima. At the same time, the normal requirements for efficient fractionation demand that the packing should distribute refluxed liquid condensate as evenly as possible over its surface, i.e., without channelling, and that it should bring about good contact between the ascending vapour and the descending condensate.

To some extent these two sets of requirements are incompatible. That is to say that the fractionating efficiency of a packing cannot be increased beyond a certain point without a commensurate increase in its resistance to gas flow and hence in its tendency to raise the operating pressure differential down a column in which it is used.

Hitherto, in distillation procedures where it has been of paramount importance to minimise the pressure drop up the column the packing used has usually consisted of a number of single sheets made of wire fabric, perforated metal or expanded metal disposed vertically in the column, i.e. with their planes parallel to the cylindrical axis of the column. These sheets are either interlocked and criss-crossed or corrugated in such a way as to form vertical tubular ducts or, alternatively, they are assembled in cylindrical sections each composed of a number of parallel, equally spaced vertical sheets, the sections being mounted, one above the other, possibly with the planes of the sheets in each section being at right angles to those of the sheets in the adjoining sections.

Such arrangements give rise to a very low pressure drop down the column during distillation, but this tends to be at the cost of some loss of fractionating efficiency because of uneven distribution of refluxing liquid condensate over the sheets. Other types of low pressure packing such as the so-called "spray pack" packing employ similar single wire fabric, perforated metal or expanded metal sheets in the form of labyrinths or honeycombs set asymmetrically to the vertical. This increases fractionation efficiency but on the other hand it also increases the column pressure drop during operation.



A type of perforated metal sheet found useful in such previous constructions is the material known as expanded metal; this is manufactured by cutting a regular matrix of parallel rows of slits in a planar sheet of metal and then applying a tension to each row in turn in the plane of the sheet in a direction at right angles to the line of the slits. In this way the sheet is expanded into a mesh comprising approximately rhombic orifices whose longer diagonals or long way mesh, are determined by the length of the original slit and whose shorter diagonals or short way mesh, are determined by the strength of the applied stress. The expansion also results in the rhombic planes of the mesh strands all being inclined at an angle to the original unexpanded sheet. Expanded metal thus has a surface extending in three dimensions which facilitates the distribution of liquid thereover.

The invention is further illustrated in the drawings wherein

FIG. 1 is a perspective view of a section of a sheet;

FIG. 2 is a perspective view of a double plate;

FIG. 3 is a diagrammatic representation of a section on the line XY of FIG. 2;

FIG. 4 is a diagrammatic exploded perspective view of three double plates;

FIG. 5 is a diagrammatic plan view of a plurality of double plates joined together to form a packing assembly; and

FIG. 6 is a diagrammatic plan view of a distillation column containing vertically alternating packing assem-

A typical expanded metal sheet is illustrated with reference to FIG. 1 of the accompanying drawings, which is a perspective view of a section of sheet comprising a number of identical orifices **1**. A long way mesh is shown by the line L and a short way mesh by the line S. Each orifice **1** is bounded by four mesh walls **2A** and **2B** and **3A** and **3B** on the upper left, upper right, lower left and lower right sides respectively. The mesh walls **2A** and **2B** constitute the strand **2** and the mesh walls **3A** and **3B** constitute the strand **3**. The strands **2** and **3** meet strands bounding adjacent holes **1** at strand junction **4**. The mesh walls do not lie in the median plane of the sheet but are inclined and slightly twisted in such a way that the lower halves **4B** of the strand junctions **4** are slightly concave and the upper halves **4A** are slightly convex, as viewed in the drawing. The strand junction **4** as a whole is disposed obliquely to the median plane of the sheet, its upper periphery being above it (as seen in the drawing) and the lower one below it.

We have now discovered a distillation column which combines a high fractionating efficiency with a low resistance to gas flow. The invention provides a packing material for a distillation column comprising a plurality of double plates, each double plate consisting of two expanded metal sheets attached to each other back to back with the strand junctions of each centrally overlying the orifices of the other and the strands of each sheet which project outwards from the median plane of the double plate being located below the orifice bounded by the said strand.

The invention further provides a still column containing as a packing material a plurality of the double plates just described. The double plates in the novel still columns may be criss-crossed and interlocking or corrugated so as to form vertical tubular ducts or may be arranged in a labyrinth or honeycomb formation as with previous types of distillation packing but they are preferably arranged in the column in vertically alternating cylindrical sections of cross sectional areas substantially equal to that of the column with each section comprising a number of vertical, parallel and equidistant double plates disposed at right angles to the plates in the adjoining sections.

The material of the expanded metal may be of any suitable metal or alloy which is wetted by the substances to be distilled. Stainless steel is suitable for many applications.

The optimum mesh configuration of the expanded metal used in the double plates will normally be similar to that for expanded metal sheets as used singly in the previous types of packings described previously but will depend generally upon the viscosity and wetting characteristics of the distillates used and upon the temperature, pressure and rate of distillation used in any given operation. We believe that the efficacy of this type of configuration in distributing a down-flow of liquid and in contacting ascending vapour therewith is largely due to the distribution of metal around the rhombic orifices on each side of the plate. Since the lower external strands bounding the orifices obtrude outwards beyond the upper, and since the internal apertures are wholly or partly blocked by the bridging strand junctions of the backing sheet the orifices can act to some extent as traps or weirs for downcoming liquid flowing over the outside of the plate along the metal strands of the mesh. Shallow pools of liquid therefore accumulate in each orifice into which liquid constantly enters and overflows at either side. The pools should be as shallow as possible so that their entire volume and not just a surface layer is continually exchanged.

Moreover, where, as is preferably the case, the strand junctions behind the orifices are somewhat smaller in area than the orifices themselves then each orifice has behind it and protruding part-way through it two or more lips comprised of the edges of the strands of the backing sheet. It is thought that the presence of these lips aids the distribution and mixing of descending liquid by allowing it to percolate between the two sheets of the plate. Also we have noted that the most efficient packings of our invenion have orifices of a size suitable to permit the downcoming liquid to form a film over as much of their areas as possible and at least over their more acute corners. These films also aid liquid distribution and vapour-liquid contact.

In the light of the above considerations we are led to believe that the principal features making for efficiency in the novel packing include strands of sufficient width to provide an appreciable surface area for liquid to flow over and orifices adapted to allow pools and films of liquid to form in and across them as described. As stated the dimensions necessary to achieve these conditions will vary with operating parameters, e.g. the surface tension and viscosity of the condensate, but we have obtained very effective liquid distribution in many systems using double plates whose sheets have short-way meshes of approximately ⅛ in., long way meshes of approximately 0.4 in. an orifice area, viewing the sheets at normal incidence, of approximately 50% of the total sheet area and an overall sheet thickness of approximately 0.01 in. Each and most particularly all, of such features are preferred. Since each row of slits in expanded metal has been expanded separately it can happen that the short way mesh sizes of the orifices vary slightly from row to row across the sheet in the direction of expansion. For this reason it is advisable to construct the double plates of two halves of a single expanded metal sheet which has been folded over and cut in a line along the short-way mesh, the two halves having been then shifted relative to each other along the same line just sufficiently to allow the meshes to interlock in the manner described.

The two sheets of the double plates may be held together in any way found suitable but as a result of their interlocking position very little extra fastening will normally be needed.

Any suitable means will normally be adequate to secure or construct the formed double plates in the chosen arrangement, which may be for example a labyrinth, a grid or alternating sections of differently aligned parallel plates as described hereinbefore. Methods presently used for securing similar arrangements of single expanded metal sheets will normally be adequate.



It is preferred to arrange the double plates in the column in vertically alternating cylindrical sections of parallel, vertical plates aligned at right angles to the plates in the adjoining sections. As illustrated in FIGS. 4, 5 and 6, in order to space the assembled plates **9** from each other, shallow bowl-shaped mounds **11** are pressed into each single sheet **5**, **5'**, such that when two sheets **5**, **5'** are interlocked to form a double plate **9**, the mounds **11** in each plate overlie each other, both pointing outwards in opposite directions. When two double plates **9** are brought together these protuberances **11** meet and space the plates **9** apart as illustrated in FIG. 5. The assembly **15A**, **15B** may then be secured simply by passing a supporting rod **12** through holes **10** drilled through the plates **9** at the centres of the protuberances **11** and fastening the external sides of the outside plates **9** in position by means of retainers **13** and **13A** around the rod. Alternatively cylindrical spacers may be used in place of the protuberances to maintain the plates at the chosen separation.

Packings of the invention are robust and will normally withstand mechanical cleaning methods, such as brushing, which many previous low pressure packings are too delicate to bear.

A double plate of the invention will now be illustrated by way of example with reference to FIGS. 2 and 3 of the accompanying drawings, of which FIG. 2 is a perspective view and FIG. 3 a diagrammatic representation of a section on the line XY.

The double plate depicted consists of two identical expanded metals sheets, **5** and **5'** of the type described previously with referance to FIG. 1, placed back to back so that the strand junctions of each are in juxtaposition to the orifices of the other. The double plate thus has a dislocated symmetry about its median plane and has the same appearance when viewed from either side.

In the drawing the sheet **5'** is behind the sheet **5**, the strand junctions **4'** of sheet **5'** being visible centrally located behind the orifices **1** of the sheet **5**. The strand junctions **4'** are somewhat smaller than the orifices **1** so that there are gaps such as **6**, **7** and **8** through the plate, caused in fact by the overlap of the orifices **1** and **1'** of the two sheets **5** and **5'**. Gaps **6** and **7** lie at either end of the long way mesh of the orifices **1** and the gaps **8** lie at the lower ends (from the point of view of the drawing) of the short way mesh.

The strand junctions **4** of the sheet **5** are inclined to the perpendicular, their upper halves **4A** being in front of the lower halves **4B**. The strand junctions **4'** of the rear sheet **5'** are similarly inclined to the perpendicular but in the opposite sense, that is their upper halves **4A'** are behind their lower halves **4B'**, as viewed in the drawing.

Thus the upper halves **4A** of the strand junctions **4** and the lower halves **4B'** of the junctions **4'** form protruding lips defining the gaps **8**. A similar arrangement exists on the reverse side of the plate from the lower halves **4B** of the junctions **4** and the upper halves **4A'** of the junctions **4'**.

The double plate consisting of expanded metal sheets **5** and **5'** is referred to as double plate **9** in FIGS. 4, 5 and 6. The double plates **9** are arranged in column **14** in vertically alternating cylindrical sections **15A** and **15B** of cross sectional areas substantially equal to that of the column **14** with each section **15A**, **15B** comprising a number of parallel vertical double plates **9** disposed at right angles to the plates **9** in the adjoining sections.

The invention is illustrated by the following example.

## EXAMPLE 1

A distillation column **14** of height 3 ft. and internal diameter 4 ins. was constructed from glass, using compressed asbestos gaskets with P.T.F.E. insets for all flanged

joints. The column was packed with 4 in. diameter, 4 in. high cylindrical packing sections, each of which consisted of a number of parallel, vertical double plates 9 of the invention with their centres evenly spaced approximately 5/16 ins. apart. The plates were maintained in position by spacers, in the form of washers, between adjacent plates. Each section was held rigid by means of two rods 12 passing through each set of spacers and plates and secured at each end by nuts 13 and washers 13A.

The double plates used were as described with reference to the accompanying drawings, FIGS. 2 and 3, and consisted of two expanded metal sheets having short-way meshes approximately 0.125 in., long way meshes of approximately 0.4 in., orifice areas, viewing the sheets at normal incidence, of approximately 50% of the total sheet area and overall thickness of approximately 0.01 in.

The sections 15A and 15B of packing were placed in the column 14 so that the planes of the double plates 9 in each were at right angles to those of the double plates in the adjoining sections.

The column described was fitted to a 20 litre flask as a reboiler which contained a solution of 10 moles percent 2-methylnaphthalene in n-decanol, both materials having a purity of greater than 98%. The reboiler was heated by an electric mantle. Reflux adaptors, fitted above and below the column section, enabled the liquid reflux to be collected and be distributed onto the packing in the case of the upper adaptor and passed through a boil-up ratemeter in the case of the lower adaptor. Thermowells were fitted to each reflux adaptor. Pressure tappings above and below the packed section were connected to an inclined manometer, and the upper tapping was also connected to a McLeod gauge, which gave the absolute pressure at the top of the packing. Two large primary coil condensers, with water at 40° C. circulating, condensed the vapour emerging from the top of the column. Water at 40° C. was necessary to prevent any 2-methyl naphthalene from solidifying on the coils. A small secondary condenser, supplied with tap water, was fitted above the primary condensers to protect the vacuum pump which was connected to the top of the secondary condenser via a reservoir.

The vacuum in the column was controlled by a fine needle valve which bled a small amount of air into the vacuum line between the pump and the reservoir.

A top sampling point was situated in the upper reflux adaptor, and a bottom sampling point in the liquid return pipe from the boil-up rate meter. A single horizontal pipe used as a spray bar turned through 90° relative to the top packing element distributed the liquid reflux to the packing.

The whole column below the condensers was insulated and the column section and upper reflux adaptor had external heating for adiabatic operation.

Nitrogen was blend into the reboiler to promote even boiling.

Before each run the packing was preflooded and then the reboiler heater adjusted to give approximately the required boil-up rate. The temperatures in the thermowells, samples from the sampling point and pressure drop and boil up rate readings were taken every half hour until equilibrium was reached, usually about 3 hours from start-up.

A number of runs were performed at various boil up rates. When equilibrium had been reached on each run the pressure drop down the column and the compositions of the tops and bottoms reflux samples were noted. The compositions were determined by comparing the refractive indices of the samples taken against a calibration of the refractive indices of a number of n-decanol/2-methylnaphthalene mixtures of known composition. Refractive index was measured using a refractometer calibrated for the mean sodium line at 5893° A. and maintained at 40° C.±0.05° C. From these measurements the number of theoretical plates in the column and hence the height equivalent to a theoretical plate (H.E.T.P.) were determined using the McCabe-Thiele relation which was taken to be applicable since the molar latent heats of the n-decanol and 2-methylnaphthalene are similar. The vapour liquid equilibrium data for these materials at 1.5 and 3 mms. Hg pressure are to be found in a paper by Taylor, Ellis and Hands in J. Appl. Chem. 16 (1966) p. 245. The pressure drop down the column per theoretical plate was also calculated. Results are shown in Table 1. The percentages free cross-sectional area in the column was approximately 69% and the column head pressure 1.5 mms. Hg.

TABLE 1

| Run | Boil-up rate (lb. moles/hr. ft.²) | Boil-up rate (lbs./hr. ft.²) | Total pressure drop down clm. (mm. Hg) | Tops composition (mole percent) methyl naphthalene | Bottoms composition (mole percent) methyl naphthalene | Number of theoretical plates | H.E.T.P. (ft.) | Pressure drop/theoretical plate (mm.Hg/T.P.) |
|---|---|---|---|---|---|---|---|---|
| 1.1 | 3.26 | 513.0 | 1.14 | 27.5 | 6.52 | 1.513 | 1.98 | 0.754 |
| 1.2 | 0.925 | 145.6 | 0.132 | 40.95 | 5.09 | 2.085 | 1.438 | 0.0632 |
| 1.3 | 1.836 | 289.0 | 0.355 | 29.7 | 5.30 | 1.7615 | 1.705 | 0.202 |
| 1.4 | 1.562 | 246.0 | 0.27 | 29.4 | 5.10 | 1.782 | 1.684 | 0.1514 |
| 1.5 | 2.344 | 369.0 | 0.617 | 25.2 | 5.0 | 1.656 | 1.812 | 0.373 |
| 1.6 | 2.46 | 387.0 | 0.656 | 26.2 | 5.7 | 1.578 | 1.90 | 0.417 |
| 1.7 | 1.224 | 192.7 | 0.1927 | 35.1 | 4.9 | 1.944 | 1.542 | 0.099 |
| 1.8 | 1.822 | 287.0 | 0.378 | 28.1 | 4.6 | 1.806 | 1.662 | 0.209 |
| 1.9 | 0.81 | 127.5 | 0.113 | 40.15 | 4.55 | 2.257 | 1.33 | 0.0497 |
| 1.10 | 1.032 | 162.6 | 0.18 | 30.65 | 3.20 | 2.2065 | 1.36 | 0.0804 |
| 1.11 | 2.404 | 378.5 | 0.625 | 19.30 | 3.45 | 1.697 | 1.767 | 0.3685 |

NOTE.—The H.E.T.P. and pressure drop per theoretical plate values compare favourably with those obtainable using alternative packings under similar conditions.

We claim:

1. A packing material for a distillation column comprising a plurality of double plates, each double plate comprising two substantially identical expanded metal sheets positioned in contact with and back to back to each other about a median plane, each of said expanded metal sheets having generally rhombic shaped orifices, each orifice being bounded by strands interconnected at strand junctions, each orifice being bounded by respective strands projecting inwardly and outwardly with respect to said median plane, said expanded metal sheets being positioned with the strand junctions of one sheet substantially centrally overlaying the orifices of the other sheet, the outwardly projecting strand portions at a junction being located with the same disposition relative to orifice boundaries for both of said sheets; at least substantially the entire confronting surface of adjacent double plates being spaced apart in the direction perpendicular to said confronting surfaces.

2. A packing material according to claim 1 wherein each double plate is constructed from a single expanded metal sheet which has been folded over and cut in a line

along the short-way mesh, the two halves having been then shifted relative to each other along the same line just sufficiently to allow their meshes to interlock.

3. In a still column containing a packing material, the improvement comprising utilizing as the packing material, a plurality of vertically positioned double plates according to claim 1 and wherein each of said outwardly projecting strand portion is located below the orifice bounded by said strand.

4. A still column according to claim 3 wherein the double plates are arranged in the column in vertically alternating cylindrical sections of cross sectional areas substantially equal to that of the column with each section comprising a number of parallel vertical double plates disposed at right angles to the plates in the adjoining sections.

5. A still column according to claim 4 including shallow bowl-shaped depressions pressed into each expanded metal sheet and protruding outwards from the plane of each double plate so as symmetrically to touch similar protuberances on the adjacent double plates, said bowl-shaped depressions having holes in the centers thereof; and a supporting rod passing through said holes in said depressions and retainers fixed around the supporting rod and touching the external sides of the outside plates of the section.




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U.S. Cl. X.R.

202—197; 261—101, 108